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MEMORANDUM REPORT ARLCB-MR 83002

EFFECTS OF CURING TEMPERATURE AND TIME ON CORROSION RESISTANCE, WEAR LIFE, ADHESION AND COMPLETENESS OF CURE OF SANDSTROM SOLID FILM LUBRICANT No. 9A

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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The effect of curing temperature and time on a heat cured solid film lubricant (SFL) were investigated using accelerated corrosion and wear tests. Surface preparation and quality control inspections were also checked. Air cured SFL's were evaluated for corrosion resistance and wear life. A compromise between corrosion resistance and wear life must be made when selecting curing temperature and time for heat cured SFL. Air cured SFL's showed very poor corrosion resistance and wear life.

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STATEMENT OF THE PROBLEM

The object of this report was to investigate the effects of curing time and temperature on Solid Film Lubricant (SFL) using accelerated corrosion and wear tests. This was prompted by reports from Aberdeen Proving Grounds of premature removal of SFL applied to the sliding surfaces of 155mm gun tubes. Other areas investigated included:

a. The optimization of the curing temperature and time of heat cured SFL to reduce manufacturing costs and maximize performance.

b. A review of the use of solvents for surface preparation and quality control inspections.

c. Evaluation of air cured SFL's for wear life and corrosion resistance.

INTRODUCTION

General data drawing #8769470 specifies the following process for the application of SFL. Surface preparation of the component will start with either hot alkaline cleaning or abrasive blast cleaning, a water rinse and then cleaning with a two part solvent consisting of 50 percent toluene and 50 percent methylethyl ketone. The component will then be spray-coated with heat curing SFL per MIL-L-46010, with a final coating thickness of between .0004 inches to .0010 inches. The SFL presently used is Sandstrom SFL #9A produced by the Sandstrom Products Co., Port Byron, Illinois. Curing of SFL #9A involves both temperature and time. The curing starts at 300°F but Sandstrom recommends 400°F for one hour in order to yield optimum corrosion protection and wear life. Sandstrom's general rule is that baking at lower temperatures will result in a slightly lower wear life but will increase

the corrosion protection. The drawing specifies heat curing at 400°F ± 10°F for 60 minutes ± 2 minutes for steel components. An important note is that the temperature refers to the temperature of the component and not just the temperature of the oven.

After the heat curing of the SFL, the informal practice is to apply Lubri-Bond 200 SFL produced by Electrofilm, North Hollywood, California, to repair any scratches or handling damage to the heat cured SFL. Lubri-Bond 200 is air cured at room temperature, dries sufficiently to handle in five minutes and is completely cured in six hours.

The two most important inspection requirements are for proper adhesion and curing of the SFL coating. The adhesion, which is of course, dependen- on surface preparation, is checked by pressing masking tape per specification PPP-T-42 onto the coated surface. Upon removal of the tape there should not be any exposed bare metal surface. The completeness of cure is checked by lightly rubbing the SFL with a clean cloth, wet with a one-to-one mixture of methlethyl ketone and methylene dichloride. The removal of the SFL all the way to the base metal indicates an incomplete cure. The repair of the SFL because of the failure of either of these two tests is not allowed. The SFL must be removed and reapplied.

APPROACH TO THE PROBLEM

An analysis of the process of applying SFL revealed two possible reasons for the problem of premature failure of the SFL - (1) improper heat curing of the SFL and (2) poor surface preparation resulting in a loss of adhesion between the SFL and metal surface.

The corrosion and wear life tests were performed on samples cured at different temperatures and times in order to establish the proper temperature and time relationship that the SFL coating must be cured at.

Other samples were first cleaned using different solvents, sprayed with SFL, cured and then subjected to a tape test to check for proper adhesion. By improving the cleaning of the metal surface, the adhesion should be increased between the SFL and the metal.

All gun tubes have a nonuniform wall thickness, hence the thinner-walled muzzle end of the tube will reach the desired temperature quicker than the thicker-walled breech end. The SFL applied to the muzzle end for corrosion resistance could be overcured while trying to obtain the correct curing temperature and time at the breech end.

Gun tubes also have a large mass which must be brought up to the temperature of the furnace before curing of the SFL can begin. This requires a significant amount of time in the furnace but the SFL must be cured in order to derive any value from its application.

The temperature and time must be optimized for economic and performance reasons. The corrosion and wear life tests performed as described above also helped to make this decision of optimizing curing temperature and time.

METHODS USED

Corrosion tests were conducted in a salt fog chamber manufactured by BEMCO, Inc., Pacoima, CA, in accordance with ASTM B-117. Steel Q panels with a dull matt finish (3" x 6") were cleaned in accordance with Federal Test Method Standard #141, Method 2011, spray coated with Sandstrom SFL #9A and

cured in an electric, forced air oven. The panels were cured at temperatures ranging from 300°F to 500°F and for times ranging from one-half to four hours. The panels were subjected to the tape test to check for proper adhesion and to the solvent test to check for proper curing of the SFL.

Wear life tests were performed on a Falex Lubricant Tester produced by Faville LeValley Corp, Chicago, IL. Tests conformed to Federal Test Method Standard #791B, Method 3807.1. The Falex test pins and V-blocks were cleaned and coated in the same manner as the steel panels for corrosion testing. The pins and V-blocks were also cured at temperatures ranging from 300°F to 500°F and for times ranging from one-half to four hours.

Solvents are used both prior to the application of SFL for surface cleaning and after to check for completeness of cure of the SFL. A review of the solvents used and their effectiveness for these two processes was performed.

Steel panels were cleaned using a mixture of toluene and MEK vs. pure MEK to check for proper surface cleaning before the application of SFL. The panels were sprayed with SFL, cured at the same temperatures and time and then given the tape test to check for adhesion.

Pure MEK was also compared with the mixture of MEK and methylene dichloride to test for completeness of cure of the SFL. Steel panels were deliberately improperly cured and then tested with the two different solvents. The amount of SFL removed was then compared.

Air cured SFL's are used to repair minor damage in the heat-cured SFL coating caused by handling of the tube after curing. The corrosion resistance and wear life of air cured SFL's were tested by applying a coating on plain and

iron phosphatized steel panels and on plain and iron phosphatized Falex pins and V-blocks. The results were compared with the results of similar tests of heat cured SFL on plain steel.

RESULTS AND DISCUSSION

a. Optimize Curing Temperature and Time:

(1) Corrosion resistance - Steel panels coated with Sandstrom SFL #9A were heat cured at seven different temperatures but for the same one hour curing time and then tested for corrosion resistance in a salt fog chamber.

The following table is the result of these corrosion tests that were performed to answer the questions of the proper curing time and temperature for cannon and also to optimize curing time and temperature for economic and performance reasons:

TABLE I. Corrosion Test - Salt Fog Chamber

Curing Temperature (°F)	Curing Time (Hrs)	Avg. Time to Failure (Hrs)
500	1	2-1/2
450	1	2-3/4
425	1	3-1/2 *
400	1	3-1/4
375	1	4-1/2 *
350	1	4-1/2
300	1	5-1/2

* These two samples were prepared separately from the others which may be the explanation for the slightly high values.

Two samples were used at each temperature to obtain average failure time.

The criteria for corrosion failure of the test panels was per MIL-L-46010, Lubricant, solid film: heat cured, corrosion-inhibiting, which is the appearance of three (3) rust spots greater than one millimeter in length, width or diameter per panel.

A second set of steel panels coated with Sandstrom SFL #9A were heat cured at the same seven (7) temperatures but at four (4) different curing times for each temperature. The panels remained in the salt fog chamber for 19 hours. The panels were then visually inspected and compared with each other to determine the degree of corrosion. The following table describes the results:

TABLE II. Corrosion Test

Curing Temperature (°F)	Curing Time (Hrs)	Word Description Corrosion Resistance after 19 Hrs.
500	1/2, 1,2,4	Very Poor
450	1/2, 1,2,4	Poor
425	1/2, 1,2,4	Poor
400	1/2, 1,2,4	Fair
375	1/2, 1,2,4	Good
350	1/2, 1,2,4	Excellent
300	1/2, 1,2,4	Good

These results are also shown in Figures 1-7. Note in these figures that there are four steel panels coated with heat cured SFL and each panel was cured at the same temperature but for a different time interval.

The general rule that the manufacturer, Sandstrom, gave for corrosion resistance holds true. The lower the curing temperature, the better the corrosion resistance. These tests verify this rule but also show that curing time has very little effect on corrosion resistance.

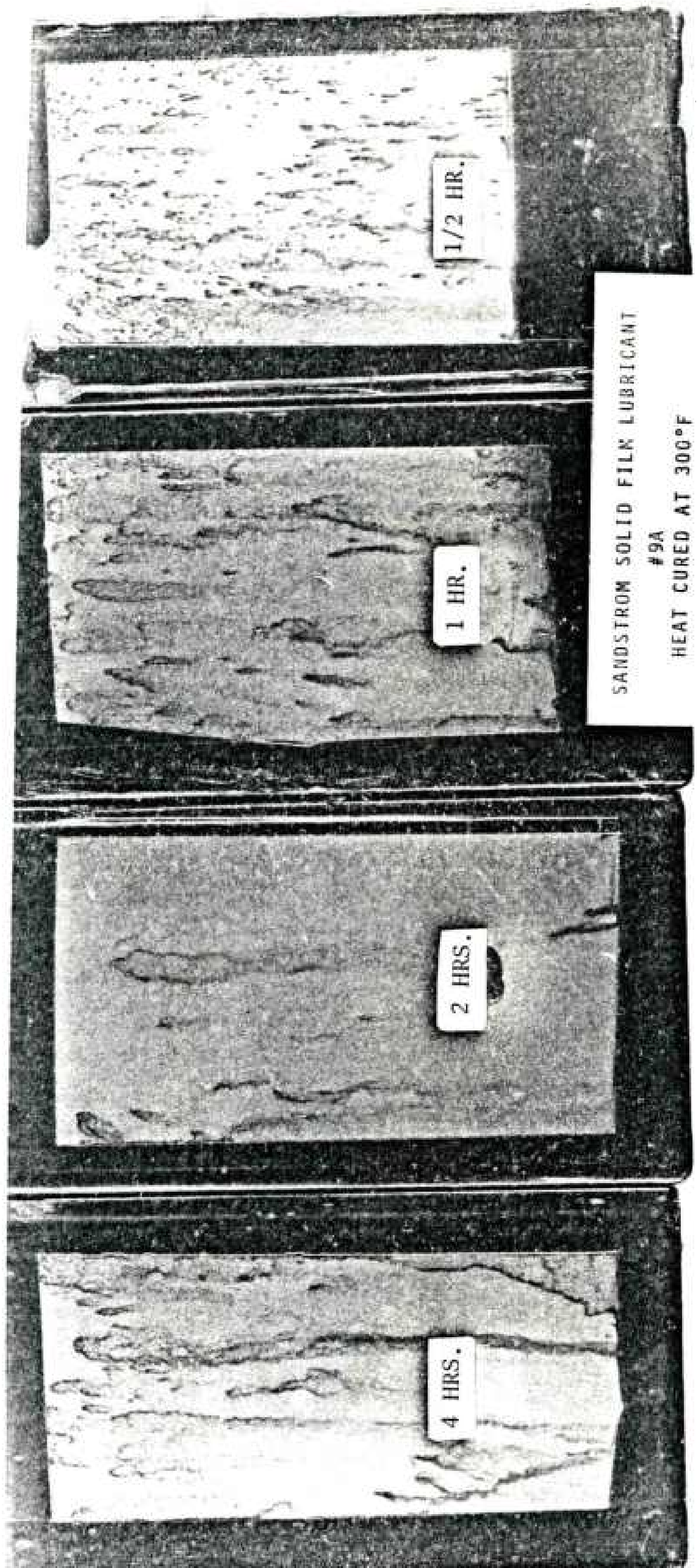
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 300°F
1/2 HR.
1 HR.
2 HRS.
4 HRS.

FIGURE 1

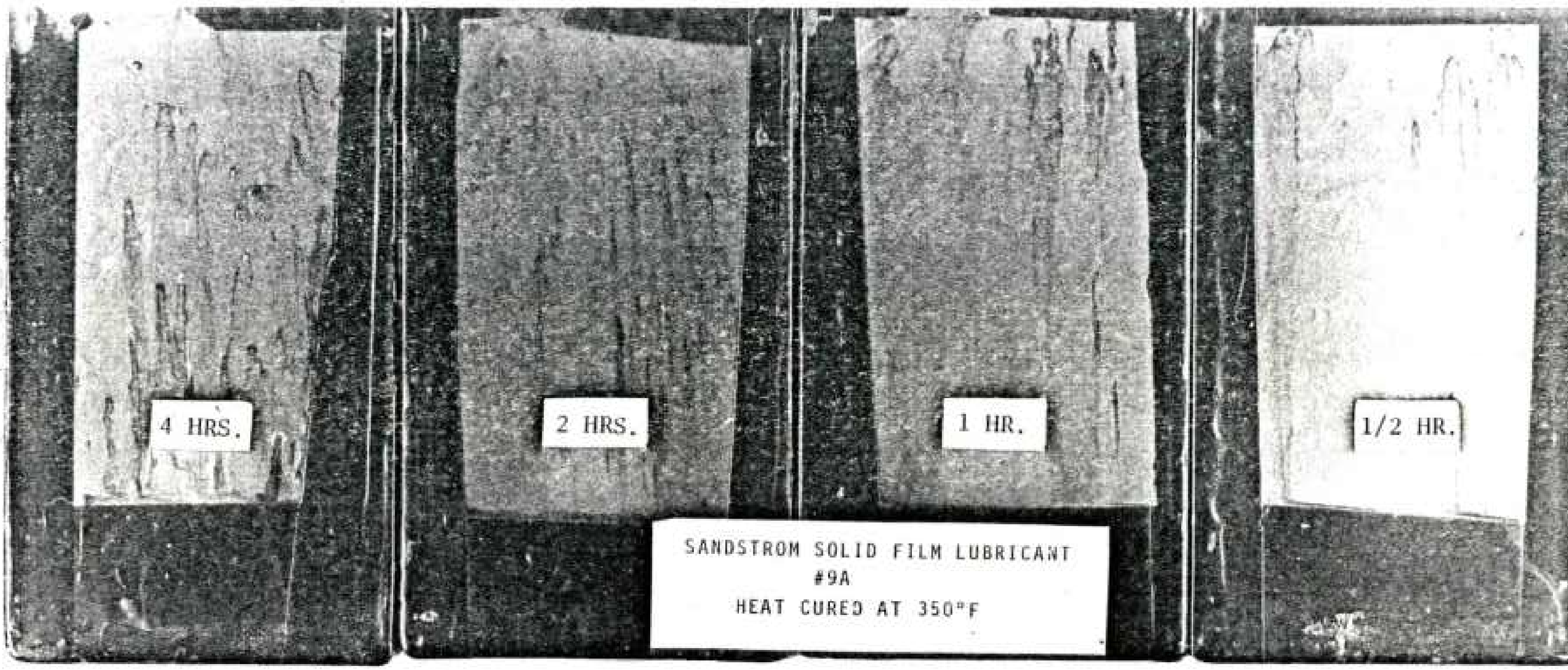
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 350°F

FIGURE 2

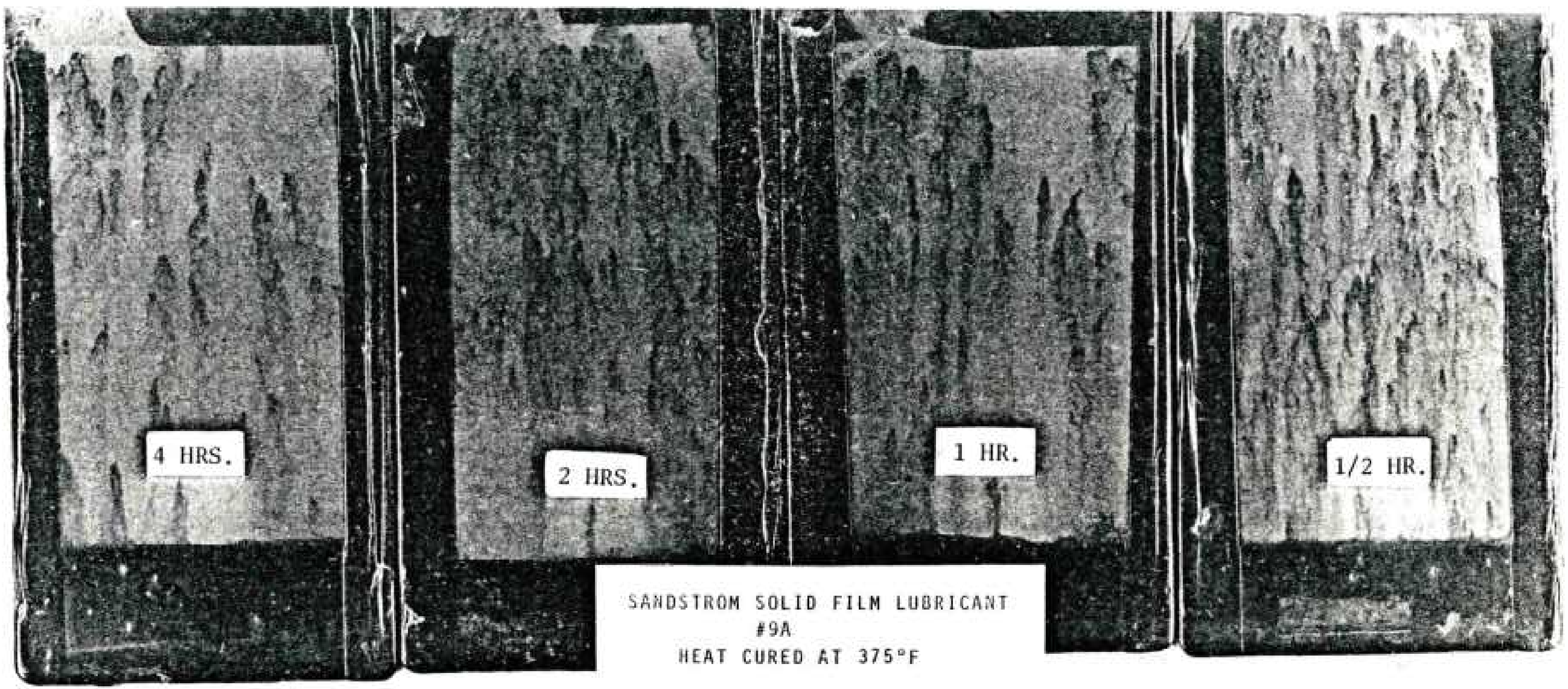
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 375°F

FIGURE 3

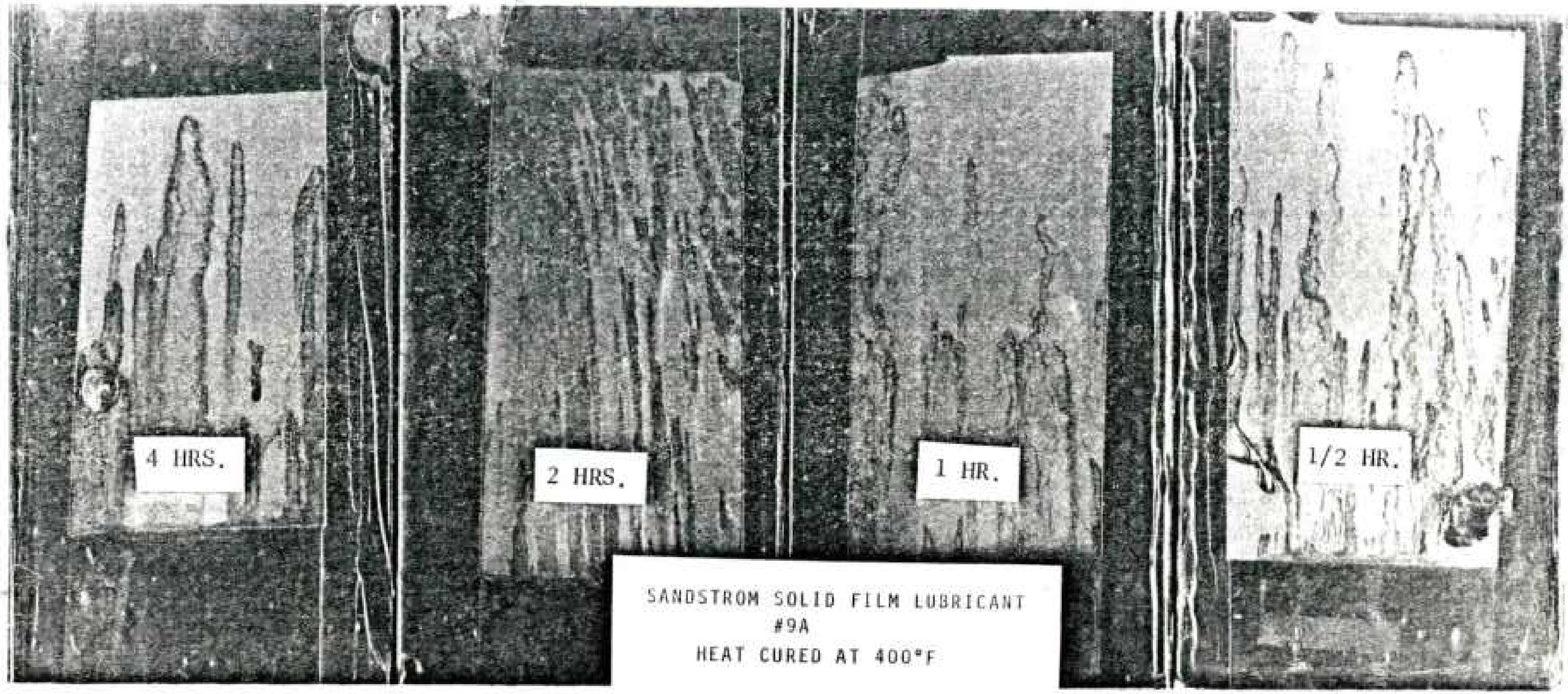
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 400°F

FIGURE 4

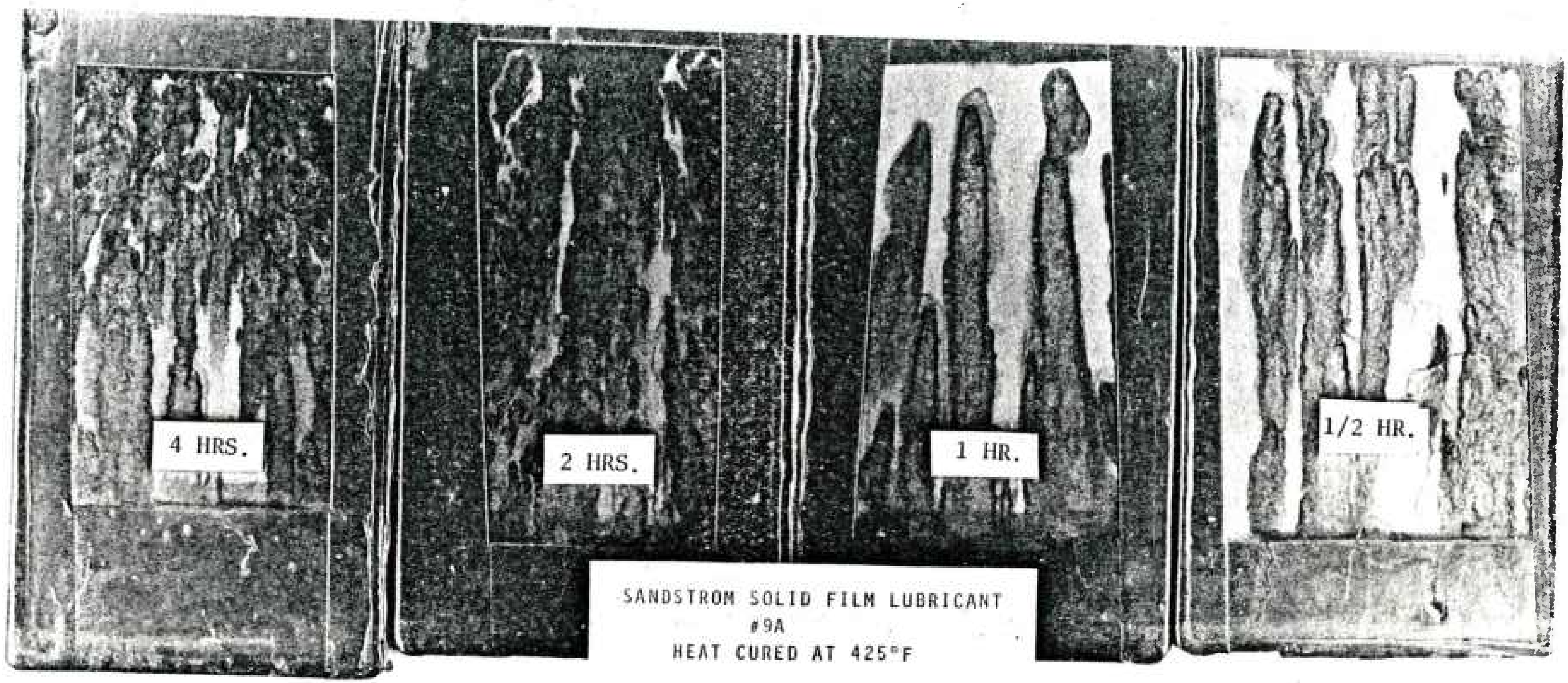
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 425°F

FIGURE 5

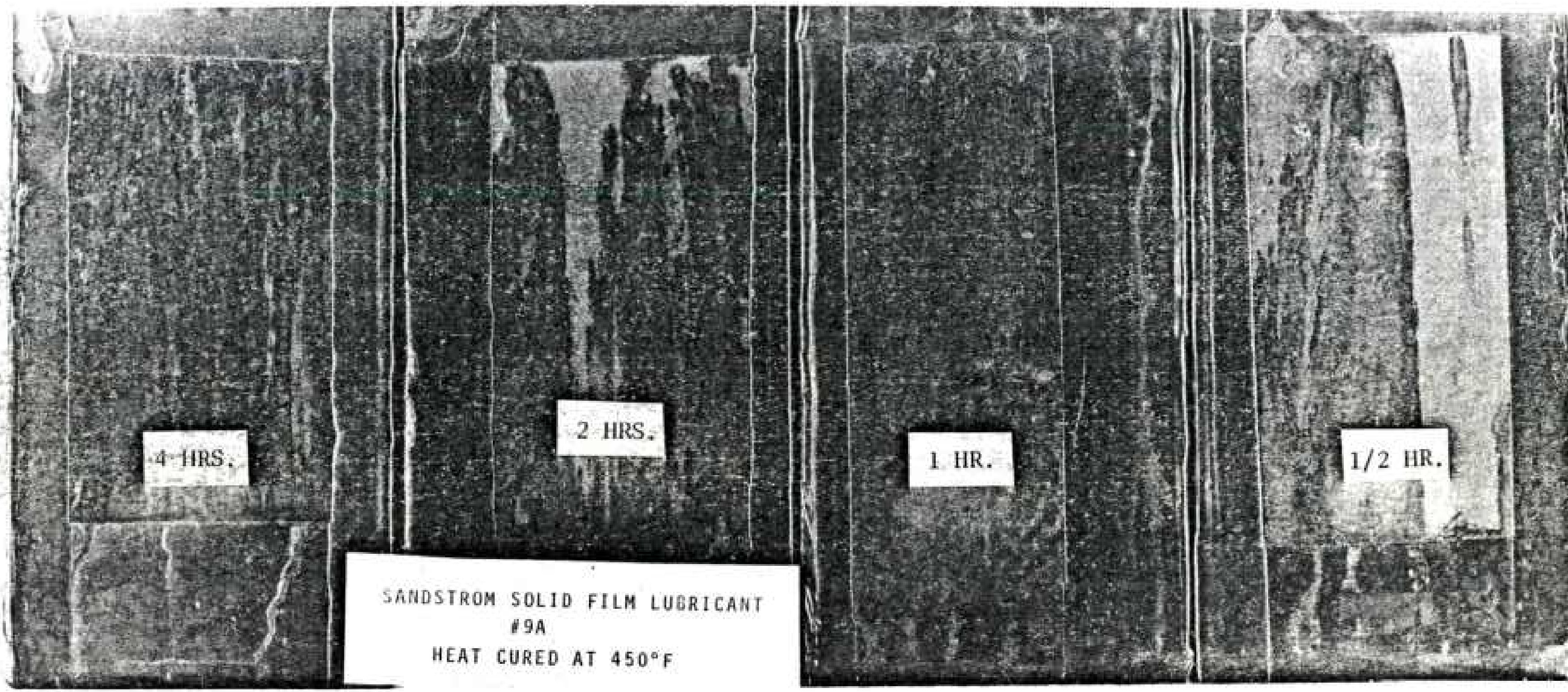
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUGRICANT
#9A
HEAT CURED AT 450°F

FIGURE 6

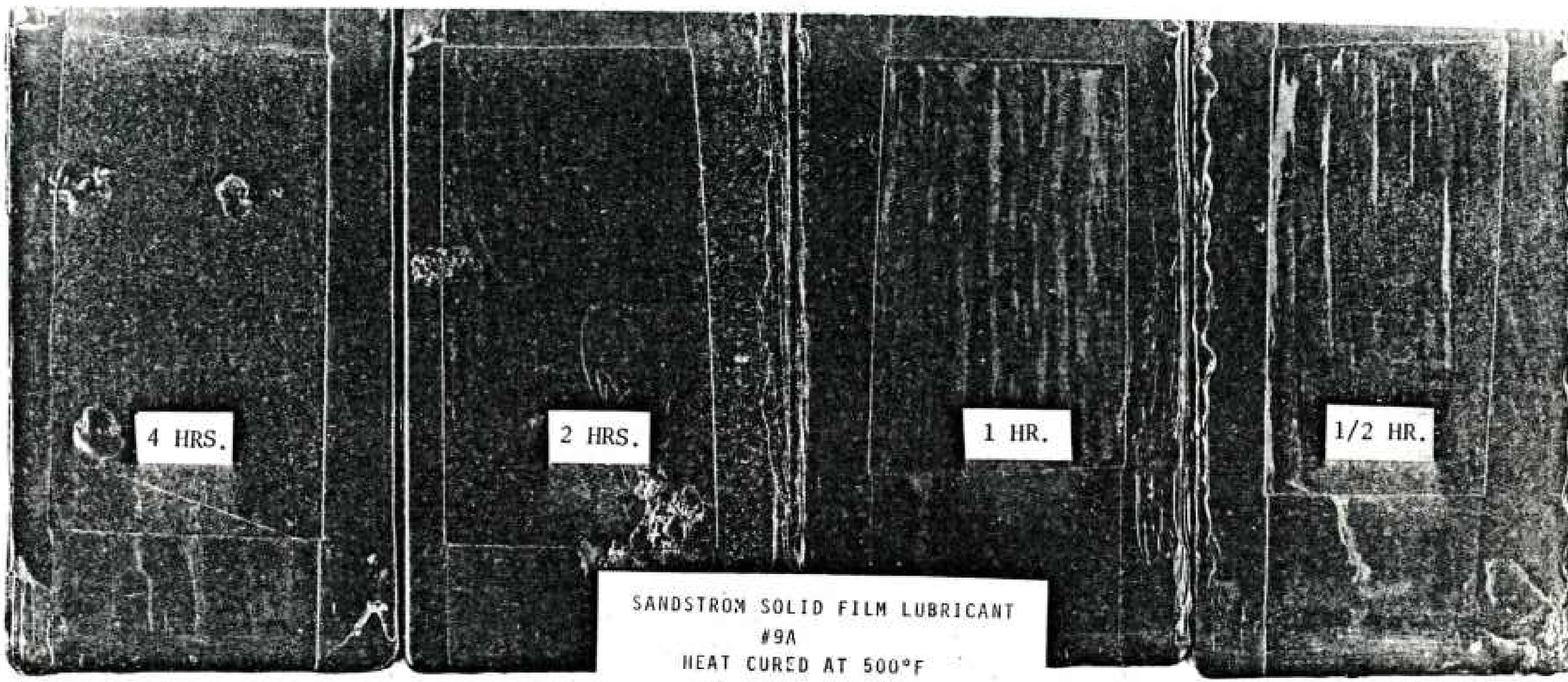
4 HRS.
2 HRS.
1 HR.
1/2 HR.
SANDSTROM SOLID FILM LUBRICANT
#9A
HEAT CURED AT 500°F

FIGURE 7

(2) Wear Life - The second major consideration when trying to optimize curing temperature and time is wear life.

Falex pins and V-blocks coated with SFL #9A were heat cured at similar temperatures and times as the steel panels used to check corrosion resistance. These samples were then tested on a Falex Lubricant tester. A summary of the results are shown in Table III.

TABLE III. Wear Life Test

Curing Temperature (°F)	Curing Time (Hrs)	Average Time to Failure (Mins-secs)
500	4	46-11
	2	50-19
	1	37-21
	1/2	30-47
450	4	14-39
	2	18-29
	1	14-41
	1/2	10-31
425	4	8-48
	2	7-14
	1	6-34
	1/2	6-36
400	4	9-08
	2	7-19
	1	6-40
	1/2	6-21
375	4	5-43
	2	5-50
	1	5-39
	1/2	5-40
350	4	6-33
	2	5-56
	1	5-35
	1/2	4-39
300	4	4-31
	2	5-24
	1	4-26
	1/2	4-28

Four samples were used at each curing time to obtain the average time to failure.

Wear life tests were made per Federal Test Method Standard 791B, Method 3807.1. Failure was indicated by a torque rise of five inch-pounds above the steady state torque value.

As curing temperature increased, so did the wear life. This was also predicted by Sandstrom but to what degree was not known until this testing was completed. Curing time had little effect on wear life at the curing temperatures of 300, 350 and 375°F. At 400° and 425°F curing time started to have a minor effect on wear life and at 450° and 500°F, curing time had a substantial effect on wear life.

TABLE IV explains the time sequence and loads used to test for wear life on the Falex Lubricant Tester.

TABLE V gives the four values of wear life and the average that resulted from these values for each time interval at each temperature.

TABLE IV. FALEX WEAR LIFE TEST PROCEDURE
(0-3000 in.-lb. Gage)

		TIME	
		To Reach or Hold Load	Cumulative Total
1.	Increase load from 0 to 300 lbs.	25 secs.	25 secs.
2.	Hold at 300 lbs.	3 mins.	3 mins.-25 secs.
3.	Increase load from 300 to 500 lbs.	20 secs.	3 mins.-45 secs.
4.	Hold at 500 lbs.	1 min.	4 mins.-45 secs.
5.	Increase load from 500 to 750 lbs.	25 secs.	5 mins.-10 secs.
6.	Hold at 750 lbs.	1 min.	6 mins.-10 secs.
7.	Increase load from 750 to 1000 lbs.	25 secs.	6 mins.-35 secs.
8.	Hold at 1000 lbs. until failure		

a. Failure is indicated by a torque rise of 5 inch-lbs above the steady state torque value or breakage of the shear pin.

b. Both pin and V-blocks are coated with SFL.

TABLE V. FALEX WEAR LIFE TEST OF SANDSTROM

SOLID FILM LUBRICANT NO. 9A

Temp(°F)	Hrs.	ID	TIME TO FAILURE MINS.-SECS. #1	#2	#3	#4	AVG.
500	4	E4	48-0	44-58	28-45	63-0	46-11
	2	E2	55-0	47-15	42-30	56-30	50-19
	1	E1	64-45	33-30	30-40	20-30	37-21
	1/2	EE	26-00	34-19	30-47	32-02	30-47
450	4	D4	13-31	13-51	15-0	16-15	14-39
	2	D2	14-58	21-12	16-52	20-54	18-29
	1	D1	14-30	9-57	13-55	20-22	14-41
	1/2	DD	12-35	13-53	8-32	7-05	10-31
425	4	N4	8-04	8-10	7-27	11-30	8-48
	2	N2	6-58	7-07	7-17	7-33	7-14
	1	N1	6-19	7-22	5-15	7-20	6-34
	1/2	NN	6-53	6-15	6-05	7-09	6-36
400	4	C4	8-37	8-20	7-50	11-43	9-08
	2	C2	7-20	6-58	7-45	7-12	7-19
	1	C1	6-45	6-17	6-50	6-46	6-40
	1/2	CC	7-03	6-10	7-06	5-06	6-21
375	4	P4	6-23	5-40	5-20	5-28	5-43
	2	P2	5-06	6-11	5-36	6-25	5-50
	1	P1	6-10	6-10	5-06	5-10	5-39
	1/2	PP	6-27	5-46	5-36	4-50	5-40
350	4	B4	6-39	6-26	6-13	6-55	6-33
	2	B2	6-05	6-09	6-15	5-15	5-56
	1	B1	5-56	5-39	5-47	4-57	5-35
	1/2	BB	4-45	4-54	4-44	4-13	4-39
300	4	A4	4-27	4-39	4-37	4-20	4-31
	2	A2	6-15	5-05	4-58	5-18	5-24
	1	A1	4-55	3-25	4-53	4-32	4-26
	1/2	AA	4-33	4-22	4-21	4-36	4-28

b. Solvents for surface preparation and inspection for completeness of cure:

The solvents used to clean the component before applying the SFL and to check the completeness of cure of the SFL after removal from the oven were tested to ensure their effectiveness.

The tape test per specification PPP-T-42 was performed on panels cleaned using a mixture of toluene and methylethyl ketone (MEK) vs pure MEK to check for proper surface cleaning and thus good adhesion. Toluene was suspect because it is produced by distilling oil and if it is not pure it could leave an oil residue on the component surface. Results of the tests indicated pure MEK was as effective in surface cleaning as a good solvent mixture of toluene and MEK. There is no need to risk using toluene with the MEK. There was no loss of adhesion when only MEK was used for surface cleaning before applying SFL.

Pure MEK was also tested against the one to one mixture of MEK and methylene dichloride to check for completeness of cure of SFL. Pure MEK was as capable of finding the deliberately uncured SFL panels as was the mixture of solvents.

c. Air dry SFL's:

(1) Corrosion resistance - Steel panels spray coated with two different air cured SFL's, Electrofilm Lubri-Bond 220 and Sandstrom 26A, were tested in the salt fog chamber to evaluate corrosion resistance. The results showed that air cured SFL's have poor corrosion resistance. They are equivalent to the results obtained with Sandstrom SFL #9A when heat cured at 450°F.

The air dry SFL's were also sprayed on iron phosphate coated panels and then tested for corrosion resistance. The iron phosphate coating increased the corrosion resistance of the air cured SFL's to equal the 400°F heat cured SFL. The iron phosphate used was a room temperature application of "SECURE LF" manufactured by DuBois Chemical Co., Cincinnati, OH.

Figure 8 illustrates the results obtained using Electrofilm Lubri-Bond 220 on plain steel and on pretreated iron phosphatized steel panels. Both panels remained in the salt fog chamber for 19 hours.

(2) Wear Life - Pins and V-blocks coated with the air cured SFL's, Lubri-Bond 220 and Sandstrom 26A, were tested on the Falex Lubricant Tester. The results showed very poor wear life for both air cured SFL's. The average failure time was 33 seconds, which means the testing load did not reach 300 lbs. The use of the room temperature applied iron phosphate (SECURE LF) undercoating did not increase the wear life.

CONCLUSIONS

a. There is no optimum overall curing temperature and time for Sandstrom SFL #9A to obtain maximum corrosion resistance and wear life. A decision must be made as to which is the most important: the higher the curing temperature, the better the wear life and the less the corrosion resistance; the lower the temperature, the better the corrosion resistance and the less the wear life. Based on the data developed in this report, the optimum curing conditions for a compromise between wear life and corrosion resistance is a curing temperature of 375°F ± 25°F and a curing time of one-half hour minimum after the component has reached temperature. However, if the component is heat cured

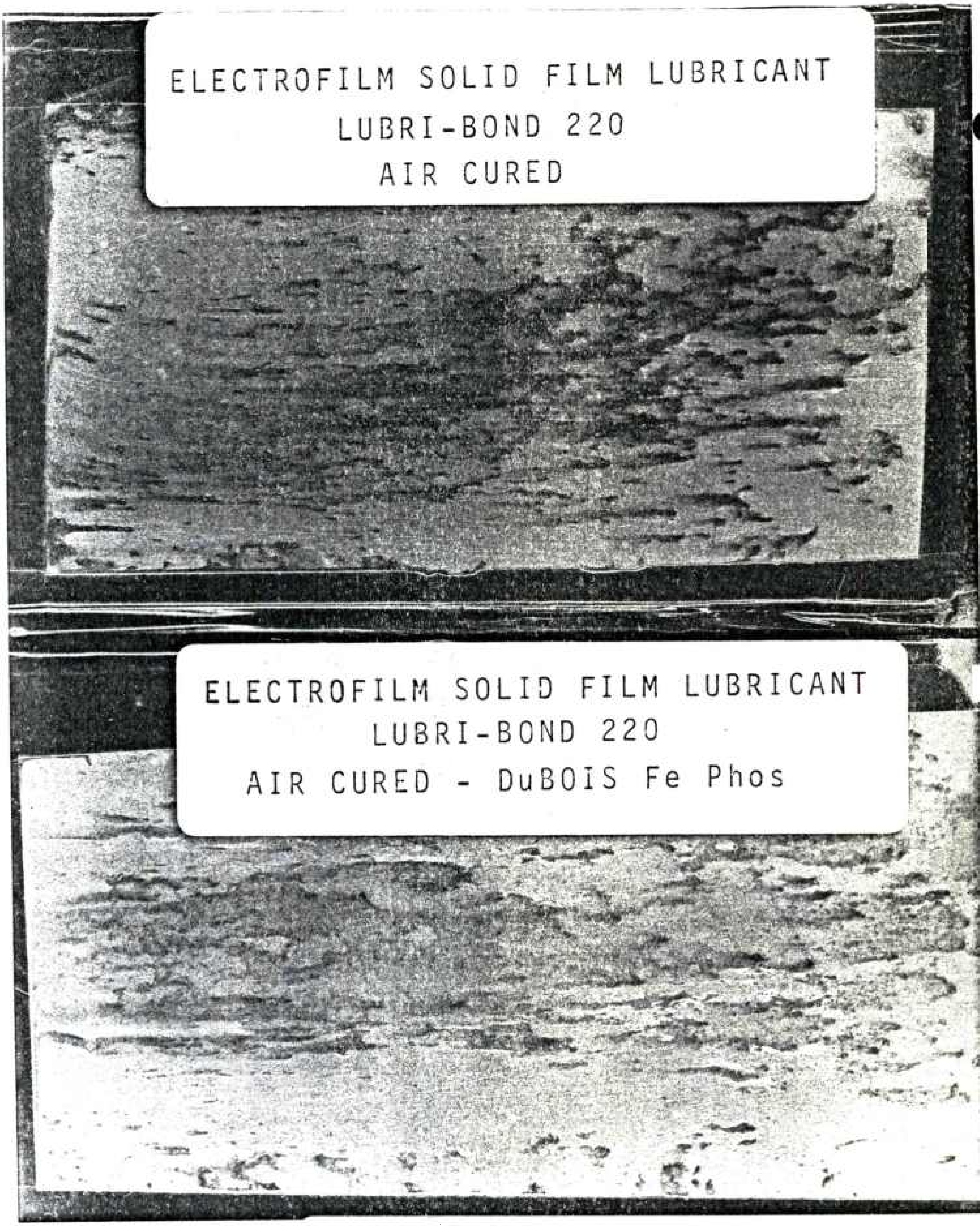
ELECTROFILM SOLID FILM LUBRICANT
LUBRI-BOND 220
AIR CURED
ELECTROFILM SOLID FILM LUBRICANT
LUBRI-BOND 220
AIR CURED - DuBOIS Fe Phos

FIGURE 8

for a longer period of time, the effect on the wear life and corrosion resistance would be negligible. Every effort should be made though to keep curing time at a minimum in order to reduce costs and manufacturing time.

b. The mixing of solvents for preparing surfaces prior to the application of SFL and for checking for completeness of cure of SFL should be eliminated. Methlyethyl ketone (MEK) is adequate for both.

c. Air dry SFL's, either Lubri-Bond 220 or Sandstrom 26A, should be restricted in their use. The poor corrosion resistance and almost non-existent wear life limit the use of these two air dry SFL's to special circumstances only.

There is no repair for improperly applied or cured SFL. If either case exists, then the SFL must be removed and reapplied.

If the component surfaces are cleaned and the SFL is cured in accordance with the General Data Drawing, the premature removal of SFL will be eliminated.

An Engineering Change Proposal (ECP) will be submitted to incorporate the changes developed in this report on General Data Drawing #8769470.

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COMMANDER NAVAL SURFACE WEAPONS CEN ATTN: TECHNICAL LIBRARY CODE X212 DAHLGREN, VA 22448	1
DIRECTOR US NAVAL RESEARCH LAB ATTN: DIR, MECH DIV	1
CODE 26-27 (DOC LIB) WASHINGTON, D.C. 20375	1
METALS & CERAMICS INFO CEN BATTELLE COLUMBUS LAB 505 KING AVE COLUMBUS, OH 43201	1
MATERIEL SYSTEMS ANALYSIS ACTV ATTN: DRXSY-MP ABERDEEN PROVING GROUND MARYLAND 21005	1

NOTE: PLEASE NOTIFY COMMANDER, ARRADCOM, ATTN: BENET WEAPONS LABORATORY, DRDAR-LCB-TL, WATERVLIET ARSENAL, WATERVLIET, NY 12189, OF ANY REQUIRED CHANGES.